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## DESCRIPTION

COLD DIE STEEL EXCELLENT IN CHARACTERISTIC OF  
SUPPRESSING DIMENSIONAL CHANGE

### TECHNICAL FIELD

The present invention generally relates to a tool material, and particularly relates to a cold die steel preferably used in a die assembly for molding components for a household electrical appliance, a mobile telephone, an automobile and the like.

### BACKGROUND OF THE INVENTION

Conventionally, JIS SKD11 has been in heavy usage for a cold die steel, but some attempts have been made to modify the SKD11 in order to improve machinability, toughness and hardness after secondary hardening. For instance, there proposed are (1) a cold die steel referred to 10%Cr SKD (see JP-A-11-279704) which has improved machinability and toughness, in which amounts of added carbon and chromium are adjusted so as to reduce non-solute carbides while the matrix composition are maintained to be that of SKD11 as much as possible, and (2) a cold die steel referred to 8%Cr SKD (see JP-A-01-011945) which has improved secondary hardening capability, in which an amount of non-solute carbides are reduced and an amount of molybdenum is increased while the matrix composition are maintained

to be that of SKD11 as much as possible.

The above described techniques are effective to improve characteristics required for a cold die steel. However, these techniques have problems to  
5 cause a large dimensional change in a tempering step. Specifically, a great dilatation in a secondary hardening region in tempering leads to increase working steps after heat treatment.

The dilatational change of dimension in  
10 tempering is caused by a release of a residual stress formed in a previous quenching step (by decomposition of retained austenite), and is promoted by precipitation of temper carbides formed by molybdenum and the like which are conventionally added expecting  
15 for secondary hardening. In addition, if the retained austenite is restrained by non-solute primary carbides which have been formed in casting and originally existed, the decomposition thereof in the tempering step is suppressed. However, it is preferable to  
20 reduce the primary carbides because they are a factor of degrading machinability, and so the reduction thereof promotes the decomposition of the retained austenite and the dimensional change.

#### SUMMARY OF THE INVENTION

25 In recent years, in processing industries of a die assembly, the number of working steps before heat treatment is sharply decreased by development in a

processing technique, but the number of working and adjusting steps after heat treatment is not so much changed. Thus, improvements in steps after heat treatment become an urgent matter in particular.

5 Accordingly, the present invention is to provide a cold die steel particularly suitable for a tool material, in which dimensional change is suppressed in quenching and tempering, so that the working and adjusting steps after heat treatment can be reduced which has increased  
10 the number of die-manufacturing steps.

First, the present inventors searched for techniques of canceling the dimensional change adversely to suppress the dimensional change which has been difficult to sufficiently suppress, under the  
15 condition that the target steel maintains all characteristics required for a cold die steel in tempering. Furthermore, they studied in detail the structural change of a matrix during tempering, and discovered that temper carbides themselves do not  
20 contribute to secondary hardening so much. Thus, they have found new means for suppressing the dimensional change and also increasing hardness, and could obtain a cold die steel having other characteristics sufficiently as well.

25 Thus, the present invention provides a cold die steel having a chemical composition described below and excellent characteristics of suppressing the dimensional change.

The cold die steel includes, by mass%, 0.7% or more and less than 1.6% of carbon (C), 0.5 to 3.0% of silicon (Si), 0.1 to 3.0% of manganese (Mn), less than 0.05% including 0% of phosphor (P), 0.01 to 0.12% of sulfur (S), 7.0 to 13.0% of chromium (Cr), one or two elements selected from the group consisting of molybdenum (Mo) and tungsten (W) amounts of which satisfy the formula:  $(Mo + (W/2)) = 0.5$  to 1.7%, less than 0.7% including 0% of vanadium (V), 0.3 to 1.5% of nickel (Ni), 0.1 to 1.0% of copper (Cu), and 0.1 to 0.7% of aluminum (Al).

The cold die steel preferably satisfies the formula by mass%:  $Ni/Al = 1$  to 3.7%. Furthermore, the cold die steel preferably satisfies the relational formulas by mass%:  $(Cr - 4.2 \times C) = 5\%$  or less; and  $(Cr - 6.3 \times C) = 1.4\%$  or more. In addition, the cold die steel preferably includes 0.3% or less of columbium (Nb).

Important features of the present invention are to suppress the dimensional change by offsetting it, which has been difficult to be basically suppressed, while the characteristics required of a cold die steel are maintained. Although the above described temper carbides have been used for secondary hardening in spite of being the factor of promoting the dilatational change of dimension in tempering, the inventors discovered that the secondary hardening capability thereof are small through a detailed study

of the hardening behavior of the cold work die steel during heat treatment as described above. The present inventors have found means for compensating the shortage of the secondary hardening capability while  
5 suppressing the dimensional change. According to the compensation means, the excellent characteristics of suppressing the dimensional change and having high hardness can be achieved without lowering necessary characteristics including machinability and abrasion  
10 resistance.

A principle of the present invention is to provide a cold die steel excellent in characteristics of suppressing the dimensional change and having high hardness, which is based on a chemical composition in  
15 which the primary carbides are reduced and the dimensional change is suppressed in an extent of satisfying the characteristics, and to which adequate amounts of nickel and aluminum are added , and besides an adequate amount of copper is added corresponding to  
20 the amounts of nickel and aluminum.

In the present invention, nickel and aluminum form intermetallic compounds precipitating in a secondary hardening region when the above described tool steel is tempered (aged), which cause a  
25 contractional change of dimension, and thereby can cancel the above described dilatation due to the decomposition of the retained austenite. It is important for the above described setoff effect to

precipitate the Ni-Al intermetallic compounds at the secondary hardening temperature of the tool steel, and the amount of copper having the effect therefor should be appropriately controlled.

5                Furthermore, with the use of a transmission electron microscope, the present inventors studied in detail the structural change of the matrix in heat treatment for high-temperature tempering in which the retained austenite decomposes and temper carbides  
10 precipitate, which particularly causes many problems of the dilatational change of dimension. As a result, the inventors confirmed that temper carbides which promote the dimensional change greatly contribute to improve abrasion resistance, but can not confirm that fine  
15 carbides are precipitated, although they have been conventionally considered to particularly contribute to secondary hardening. They found that the degree of secondary hardening is greatly affected by factors in the matrix.

20                Ni-Al intermetallic compounds adopted in the present invention have a secondary hardening effect as a precipitation strengthening element, thereby further compensates the secondary hardening in addition to the above described setoff effect of the dimensional  
25 change. Thus, excellent resistance to the dimensional change and high hardness characteristics can be achieved without lowering other necessary characteristics such as machinability and abrasion

resistance.

The precipitation strengthening method with intermetallic compounds has been conventionally often applied to a maraging steel, but has not been used in the field of a tool steel including 0.2 mass% or more of carbon, and particularly in the field of a cold die steel which relates to the present invention. In addition to the characteristics of canceling a dimensional change, the present inventors knew that temper carbides do not actually have such a high secondary hardening effect on the tool steel in itself as has been considered, and consequently noticed to use such an intermetallic compound. Because nickel and aluminum act individually to lower the required properties of the tool steel, an appropriate composition and alloy design for the tool steel are necessary in consideration of the mutual interaction of them with copper.

Next, a dimensional change generated in quenching will be described. The degree of the dimensional change depends on an amount of a solid solute carbon existing in a matrix when it is quenched. In other words, the dimensional change is generated due to the expansion of crystal lattices by the force of a solute carbon in a martensitic structure. In a conventional steel, an alloy composition is totally designed so that the amount of solid-solute carbon can be in the vicinity of 0.6 (mass %) when quenched, which

value is analogous to SKD11. But the cold die steel according to the present invention is designed to have such a composition that the amount of solid-solute carbon is intended to be about 0.53% which is lower  
5 than that in the conventional steel.

The design of the composition is achieved by addition of elements such as copper, nickel and aluminum, which decrease the amount of solid-solute carbon. That is a principle of the design for  
10 suppressing dilatation in quenching. A preferable conditions of achieving such an amount of solid-solute carbon are, in addition to the basic composition and the appropriate amount of copper, nickel and aluminum to be added which are specified in the present  
15 invention, to control amounts of carbon and chromium to be added in the whole cold die steel so as to satisfy the formulas:  $(Cr - 4.2 \times C) = 5$  or less, and  $(Cr - 6.3 \times C) = 1.4$  or more, preferably  $(Cr - 6.3 \times C) = 1.7$  or more.

FIG. 1 shows a summarized concept for the  
20 above conditions. (\*Note: in FIG. 1, reference character A shows "an effect for suppressing dilatation due to decrease of an amount of solid-solute carbon"; reference character B shows "that the dimensional change are setoff due to precipitation strengthening";  
25 and reference character C denotes "a secondary hardening temperature of the steel according to the present invention.")

The figure shows that the cold die steel



according to the present invention causes larger secondary hardening than JIS SKD11, but can suppress the dimensional change more effectively than that. A principle of the present invention is to simultaneously  
5 satisfy the two points of: (1) decreasing the amount of solid-solute carbon in quenching (see the reference character A in FIG. 1); and (2) offsetting a volume change of a matrix in secondary hardening, due to addition of copper, nickel and aluminum (see the  
10 reference character B in FIG. 1). Regarding the concept for the item (1), it is most important industrially to control the amount of solid-solute carbon to be approximately 0.53% at a general-purpose quenching temperature of about 1,030°C. Regarding the  
15 concept for the item (2), as it is concerned that adding of copper and nickel might degrade hot and cold workabilities, it is important to keep a balance between the level for preventing the degradation thereof and that for causing the maximum precipitation  
20 strengthening.

The composition of the cold die steel according to the present invention will be explained below. In the following description, a notation of % means mass percent, for indicating a content of each  
25 element.

Carbon (C) is an important element which partly solutes in a matrix to impart strength thereto, and partly forms carbides to improve wear resistance

and resistance to seizure. A ratio of an amount of solid-solute carbon to that of carbon in carbides in the steel is mainly determined by an interaction between carbon and chromium, so that the carbon content must be simultaneously specified with chromium content in consideration of the interaction therebetween. However, in order to obtain a practical cold die steel which have well-balanced machinability and heat treatment deformation stability, the amount of carbon is independently in a range of 0.7 to 1.6%, preferably in a range of 0.9 to 1.3%.

Silicon (Si) is an important element for the cold die steel according to the present invention. Although about 0.3% of silicon is normally added as a deoxidizing agent, 0.5% or higher of silicon is added in the present invention which is higher than usual. It is important to prevent a softening of the steel below a tempering temperature of about 490°C, because it is concerned that quenching hardness of the steel may be lowered due to a design of the composition for suppressing the dilatation in quenching. However, as an excessive silicon content causes formation of a delta ferrite, the upper limit is defined to be 3.0%. A preferable amount of silicon is in a range of 0.9 to 2.0%.

Manganese (Mn) is used for a deoxidizing agent as well as silicon, and the steel contains at least 0.1% of manganese. However, as an excessive

manganese content lowers machinability, the upper limit is defined to be 3.0%. A preferable amount of manganese is in a range of 0.1 to 1.0%.

Chromium (Cr) is an indispensable element for  
5 forming carbides as well as for enhancing  
hardenability. A ratio of an amount of solute chromium  
with respect to that of chromium in carbides is  
determined by interaction between chromium and carbon  
as in the case of carbon. Thus, the chromium content  
10 must be simultaneously specified with the carbon  
content in consideration of the interaction  
therebetween. However, in order to obtain a practical  
cold die steel which has well-balanced machinability  
and heat treatment deformation stability, an amount of  
15 chromium is independently in a range of 7.0 to 13.0%,  
preferably in a range of 8.0 to 11.0%.

Molybdenum (Mo) and tungsten (W) impart a  
similar working-effect to the steel, the level of which  
can be specified by  $(Mo + (W/2))$  in consideration of an  
20 atomic weight. Molybdenum and tungsten are considered  
to play an important role in secondary hardening of a  
tool steel, so that large amounts of molybdenum and  
tungsten are added to a high-speed tool steel requiring  
high hardness, for they are used in small products such  
25 as a byte or a drill. In the present invention as  
well, molybdenum and tungsten are indispensably added  
because they greatly contribute to a matrix condition  
for developing secondary hardening. If the content is

less than 0.5% a sufficient effect is not obtained. But on the other hand, excessive addition is not preferable for large products such as a cold die assembly because these elements promote the dimensional  
5 change as described above. Accordingly, in the cold die steel according to the present invention, an amount of (Mo + (W/2)) is defined to be in a range of 0.5 to 1.7%, preferably in a range of 0.75 to 1.5%.

Aluminum (Al) is coupled with nickel to form  
10 a Ni-Al intermetallic compound such as  $\text{Ni}_3\text{Al}$  or  $\text{NiAl}$ , and causes secondary hardening through its precipitation. A matrix is retracted through the precipitation reaction, which cancels the dilatation reaction in the tool steel in secondary hardening. As  
15 a result, the dimensional change is suppressed. Thus, aluminum is an important element in the present invention. However, the aluminum content of less than 0.1% does not provide a sufficient effect, but on the other hand, an excessive content exceeding 0.7% causes  
20 a remarkable formation of a delta ferrite, so that an amount of Aluminum is specified to be in a range of 0.1 to 0.7%, preferably in a range of 0.1 to 0.5%, further preferably in a range of 0.15 to 0.45%.

Nickel (Ni) is coupled with aluminum to form  
25 a Ni-Al intermetallic compound which precipitates as described above, and simultaneously thereby causes secondary hardening and the suppression of the dimensional change. Thus, nickel is an important

element in the present invention. In addition, it is an useful element for preventing red brittleness of the cold die steel according to the present invention which contained copper as described below. The nickel  
5 content of less than 0.3% does not provide a sufficient effect, but on the other hand, an excessive content exceeding 1.5% increases a solubility limit of carbon in iron and lowers the workability in an annealed state. Thus, an amount of nickel is specified to be in  
10 a range of 0.3 to 1.5%, preferably in a range of 0.4 to 1.5%, further preferably in a range of 0.5 to 1.3%.

Furthermore, if amounts of nickel and aluminum are adjusted so as to satisfy the relationship of  $\text{Ni/Al} = 1$  to 3.7, amounts of nickel and aluminum in  
15 the matrix, which do not form an intermetallic compound, can be adjusted. Thus, the amount of nickel in the matrix can be reduced particularly after the intermetallic compound has been precipitated, so that the steel can keep good machinability after heat  
20 treatment (aging). A preferable ratio of nickel to aluminum is 1.2 to 3.7. A more preferable one is in a range of 1.3 to 3.7, and a further preferable one is in a range of 2.5 to 3.5.

As for copper, a metal phase of copper starts  
25 to precipitate from a temperature of about 480°C or higher and forms a nucleus of precipitation for an intermetallic compound, so that it enables the above described Ni-Al intermetallic compound to precipitate

just at the vicinity of a secondary hardening temperature of the tool steel, although the compound originally precipitates at a higher temperature. Accordingly, it makes the tool steel according to the present invention develop fully the offset effect of the dimensional change and the secondary hardening effect due to the precipitation of the Ni-Al intermetallic compound. However, as a large amount of copper causes red brittleness, it is important in the present invention to specify the amount thereof to be in a range of 0.1 to 1.0%, preferably in a range of 0.2 to 0.8%.

Sulfur (S) is an useful and indispensable element for the cold die steel according to the present invention, because it improves machinability. However, as an excessive content lowers toughness, the content is in a range of 0.01 to 0.12%, preferably 0.03 to 0.09%.

Columbium (Nb) is a preferable element to be contained for the cold work die steel according to the present invention, because it functions to make carbides distribute uniformly in a structure and reduce deformation due to heat treatment. An amount of columbium is preferably 0.03% or more, but is desirably 0.3% or less, because columbium contained in the steel forms an MX compound and an excessive amount of the compound impairs machinability.

In addition, the following elements may be

contained in the steel according to the present invention so far as being controlled in the following range.

Phosphor (P) is controlled to be less than  
5 0.05%, preferably 0.02% or less, because it is an element which lowers toughness. Vanadium (V) can be added to improve hardenability, but it lowers machinability. Thus an amount of vanadium is limited to be less than 0.7%, preferably 0.5% or less, even  
10 though it is added.

The present invention provides a cold die steel which satisfies the above described conditions and contains iron (Fe) substantially as the balance. Characteristics of suppressing a dimensional change and  
15 excellent secondary hardening are achieved by the cold die steel which consists of, for instance, the above described elements, iron, and other elements of 20% or less, 10% or less, or 5% or less in total, or which consist of the above described elements and the balance  
20 of iron with unavoidable impurities.

Examples according to the present invention will be described below with reference to the drawings.

#### BRIEF DESCRIPTION OF DRAWINGS

FIG. 1 is a view which shows dimensional  
25 changes and hardness due to tempering for cold die steels to explain an effect of the present invention;

FIG. 2 is a view showing dimensional changes

of cold die steels due to heat treatment;

FIG. 3A is a front view of a test piece used in an example according to the present invention, for measuring a quantity of torsion of cold die steels due to heat treatment;

FIG. 3B is a side view of a test piece used in an example according to the present invention, for measuring a quantity of torsion of cold die steels due to heat treatment; and

FIG. 4 is a view showing a quantity of torsion of cold die steels due to heat treatment.

#### DETAILED DESCRIPTION OF THE INVENTION

##### Example 1

Samples No. 1 to 6 according to the present invention and comparative examples No. 7 to 9 were melt with a high-frequency induction heating device in the ambient atmosphere, and formed into ingots with a cross section size of 80 x 80 mm , each of which has a composition shown in Table 1 with the balance of iron and unavoidable impurities. Here, the sample No. 7 is a material referred to JIS SKD11, while the sample No. 8 is 8%Cr SKD and the sample No. 9 is 10%Cr SKD.



Table 1

	Composition (mass%)														Mo+ W/2	Ni/Al	Cr- 4.2C	Cr- 6.3C
	C	Si	Mn	P	S	Cr	Mo	W	V	Ni	Cu	Al	Nb	Fe				
Present Invention No. 1	1.11	1.31	0.58	<0.01	0.06	8.95	1.1	<0.1	0.25	0.78	0.42	0.27	<0.03	Bal.	1.1	2.889	4.288	1.957
" No. 2	1.19	1.49	0.62	<0.01	0.12	9.12	0.8	0.21	0.35	1.02	0.78	0.31	<0.03	Bal.	0.905	3.290	4.122	1.623
" No. 3	0.72	1.51	0.61	<0.01	0.06	7.20	1.0	0.13	0.21	0.81	0.49	0.28	<0.03	Bal.	1.065	2.893	4.176	2.664
" No. 4	1.51	1.52	0.64	<0.01	0.01	12.3	1.25	0.32	<0.1	1.02	0.61	0.29	<0.03	Bal.	1.410	3.517	5.958	2.787
" No. 5	1.18	1.05	0.57	<0.01	0.06	10.4	0.88	0.24	<0.1	0.79	0.54	0.26	<0.03	Bal.	1.000	3.038	5.444	2.966
" No. 6	1.02	1.51	0.56	<0.01	0.07	8.40	0.91	<0.1	<0.1	0.52	0.39	0.35	0.13	Bal.	0.91	1.486	4.116	1.974
Comparative Example No. 7	1.49	0.35	0.38	<0.01	<0.01	12.2	1.04	0.35	0.25	<0.01	<0.01	<0.01	<0.03	Bal.	1.215		5.942	2.813
" No. 8	0.98	1.11	0.41	<0.01	<0.01	7.95	1.98	0.30	0.25	<0.01	<0.01	<0.01	<0.03	Bal.	2.130		3.834	1.776
" No. 9	1.18	0.39	0.42	<0.01	0.06	10.2	1.02	0.25	0.25	<0.01	<0.01	<0.01	<0.03	Bal.	1.145		5.244	2.766

At first, these ingots were hot-worked into a wire material with a cross section size of 15 mm × 15 mm. They were then annealed, and test pieces of 8 mm $\phi$  × 80 mmL were formed, and the longitudinal dimensions thereof were measured. Then, they were quenched at 1,030°C (nitrogen cooling at a pressure of 0.506 MPa), and subsequently subjected to high-temperature tempering twice to be secondary hardened so as to have a hardness around 60 to 63 HRC. The dimensions were measured again at this condition. The sample No. 8 (8%Cr SKD) is secondary hardened at a tempering temperature of about 525°C, and the other samples are secondary hardened at a tempering temperature of about 510°C. All samples of No. 1 to 6 after heat treatment have higher hardness than that of SKD11 (No. 7), which means that those samples have excellent secondary hardening capability.

FIG. 2 shows a dimensional change of each sample due to heat treatment, or, a dimensional change during secondary hardening. The dimensional change during heat treatment was calculated from the measurements on longitudinal dimensions before and after the above described heat treatment, by using the following formula.

$$\text{dimensional change during heat treatment} = \frac{(\text{dimension after heat treatment} - \text{dimension before heat treatment})}{\text{dimension before heat treatment}} \times 100$$

The sample No. 8 has the largest dilatation and shows the largest dimensional change. This is because it contains excessive molybdenum. The samples No. 7 and 9 have compositions adequately adjusted to have an amount of molybdenum equivalent of  $(Mo + (W/2))$  being about .1.0%, but still cause the dilatation around 0.05%. In contrast, the samples of No. 1 to 6 containing the appropriate amounts of nickel, copper and aluminum show the dimensional change due to heat treatment controlled to be 0.01% or less. The result shows that a precipitation reaction of Ni-Al intermetallic compounds in a secondary hardening region offsets the dilatation.

#### Example 2

A test piece having a form shown in FIGS. 3A (front view) and 3B (side view) was produced from the annealed steels. In FIG. 3A, each clearance (gap size) at the positions of arrow (1) (at 2.5 mm from the left), arrow (2) (at 5.0 mm from the left) and arrow (3) (at 7.5 mm from the left) is 0.5 mm. The test pieces were then heat-treated in the same way as in Example 1, and the clearances at the same positions were measured again. A "quantity of torsion" was determined from changes of the clearances based on the following formula;

quantity of torsion (absolute value) =  
| (average variation of (1) to (3)) - (the further value  
from the above described average value, selected from  
values (1) and (3)) |.

5           The results of the calculated quantities of  
torsion are shown in FIG. 4. The sample No. 7 has the  
largest quantity of torsion. This is because the  
sample contains a large amount of solid-solute carbon  
in the martensite and a large amount of non-solute  
10 carbide, and thereby a strong inner stress is generated  
by the dilatation of the matrix and the restraint by  
the non-solute carbides. The samples No. 8 and 9 have  
a large quantity of torsion in spite of containing few  
non-solute carbides, but the samples of No. 1 to 6, in  
15 which the inner stress of the matrix is offset by the  
precipitation of Ni-Al intermetallic compounds,  
obviously have small quantities of torsion.  
Furthermore, the sample No. 6 containing the  
appropriate amount of columbium provides an adequate  
20 result that torsion was not measured within the  
measurement accuracy of  $\pm 0.0001$  mm.

The tool steel in the present invention are  
not dimensional changed and deformed due to heat  
treatment, and makes it possible to reduce/omitted  
25 finish working for correction after heat treatment.  
Thus, manufacturing cost for the die assembly can be  
reduced. The tool steel further make it possible to

shorten a period for manufacturing the die assembly, and to heat treat a die having a more complicated shape, so that the present invention is an industrially extremely useful technology.

## 5 INDUSTRIAL APPLICABILITY

The cold die steel according to the present invention is preferably used as a tool material for forming components in machines.